Crystal structure of *N*-[4-bromo-2-(2-chlorobenzoyl)phenyl]-2-chloro-acetamide, $C_{15}H_{10}BrCl_2NO_2$

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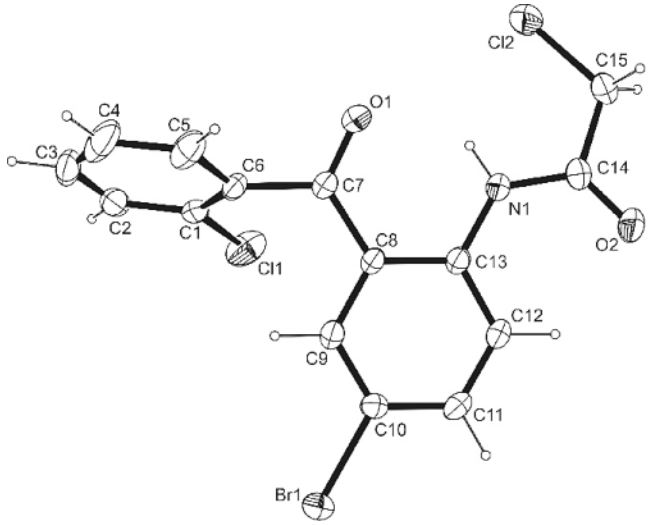

Abstract

$C_{15}H_{10}BrCl_2NO_2$, triclinic, $P\bar{1}$ (no. 2), a = 7.5922(3) Å, b = 10.0972(4) Å, c = 10.7565(5) Å, α = 69.991(1)°, β = 76.768(1)°, γ = 70.646(1)°, V = 724.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.0282, $wR_{ref}(F^2)$ = 0.0883, T = 200 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.38×0.42×0.47 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	32.06 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	56.34°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11171, 3388
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3197
$N(param)_{refined}$:	194
Programs:	SHELX [6], ORTEP-3 [7], MERCURY [8], PLATON [9]

Source of material

The title compound was obtained as a gift sample from R. L. Fine Chem., Bengaluru, India. The compound was recrystallized from a 1:1 mixture (*v*/*v*) of dichloromethane and acetone by slow evaporation at room temperature.

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å for aromatic carbon atoms and C–H 0.99 Å for methylene groups) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 U_{eq}(C). The nitrogen-bound H atom was located on a difference Fourier map and refined freely.

Discussion

Benzophenone and related compounds have been reported to show antiallergic, anti-inflammatory, antiasthmatic, antimalarial, anti-microbial and antianaphylactic activity [1]. The title compound is an intermediate in the synthesis of certain anxiolytic, anticonvulsant and sedative drugs, and is also a starting material for the synthesis of diazepam and other benzodiazepines. The crystal structures of some related compounds such as *N*-(2-benzoyl-4-chlorophenyl)-2-chloro-acetamide [2] and *N*-[4-chloro-2-(2-chlorobenzoyl)-phenyl]-acetamide [3] have been reported. In view of the importance of the title compound and in connection with our ongoing interest in pharmaceutical active molecules, we have determined its crystal structure. While one of the two phenyl groups of the benzophenone core bears only a chloro substituent in *ortho*-position, the other possesses a bromo substituent in *meta*-position as well as an amide-derived substituent in *ortho*-position. The amide moiety features a terminal chloromethyl group with the chlorine atom nearly in-plane with the nitrogen atom. The respective N–C–C–Cl dihedral angle is found at only -5.9(2)°. The least-squares planes defined by the respective carbon atoms of both aromatic substituents intersect at an angle of 66.79(11)° (Fig.). In the crystal, the nitrogen-bound hydrogen atom forms a bifurcated, exclusively intramolecular hydrogen bond with the oxygen atom of the benzophenone core as well as the chlorine atom. The latter finding might explain the small N–C–C–Cl dihedral angle as mentioned above. In addition, a C–H···O as well as a C–H···Cl contact are apparent. While the former one is supported by one of the hydrogen atoms of the chloromethyl group, the latter involves the hydrogen atom in *ortho*-position to the amide-substituent. These contacts connect the molecules to chains along [110]. Additonally, an intramolecular C–H···O hydrogen bond can be observed between the hydrogen atom that is already part of the C–H···Cl contacts and the oxygen atom of the amide moiety. In terms of graph-set analysis [4, 5], the descriptor for these contacts is $S(5)S(6)S(6)R^2_2(8)R^2_2(16)$ on the unary level. The shortest inter-centroid distance between two aromatic systems was found at 3.8586(14) Å and is apparent between the bromo-substituted phenyl moiety and its symmetry-generated equivalent.

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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	2*i*	0.088(4)	0.700(3)	0.311(3)	0.030(7)
H(2)	2*i*	0.7093	0.2051	0.0004	0.048
H(3)	2*i*	0.4998	0.1295	–0.0697	0.060
H(4)	2*i*	0.1818	0.1939	0.0057	0.062
H(5)	2*i*	0.0644	0.3435	0.1479	0.044
H(9)	2*i*	0.4262	0.2227	0.3933	0.026
H(11)	2*i*	0.3949	0.3353	0.7256	0.033
H(12)	2*i*	0.2159	0.5709	0.6290	0.031
H(15A)	2*i*	–0.0203	1.0186	0.3565	0.034
H(15B)	2*i*	–0.2059	0.9666	0.3827	0.034

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	2*i*	0.57256(3)	0.06649(2)	0.64813(2)	0.0342(1)	0.0230(1)	0.0270(1)	–0.00369(9)	–0.00989(9)	–0.00386(9)
Cl(1)	2*i*	0.66808(8)	0.37903(9)	0.16270(6)	0.0269(3)	0.0689(5)	0.0327(3)	–0.0143(3)	–0.0031(2)	–0.0157(3)
Cl(2)	2*i*	–0.0396(1)	0.94890(8)	0.17969(6)	0.0674(4)	0.0314(3)	0.0300(3)	0.0078(3)	–0.0123(3)	–0.0062(2)
O(1)	2*i*	0.1210(3)	0.5832(2)	0.1901(2)	0.0445(9)	0.038(1)	0.0250(8)	0.0080(8)	–0.0149(7)	–0.0143(7)
O(2)	2*i*	0.0194(3)	0.7905(2)	0.5594(2)	0.048(1)	0.0285(9)	0.0277(8)	–0.0058(7)	–0.0038(7)	–0.0151(7)
N(1)	2*i*	0.1083(3)	0.6829(2)	0.3898(2)	0.0284(8)	0.0211(9)	0.0196(8)	–0.0037(7)	–0.0037(6)	–0.0085(7)
C(1)	2*i*	0.5089(3)	0.3189(2)	0.1180(2)	0.032(1)	0.024(1)	0.0163(8)	0.0002(8)	–0.0061(7)	–0.0046(8)
C(2)	2*i*	0.5785(4)	0.2326(3)	0.0309(2)	0.058(2)	0.029(1)	0.019(1)	0.008(1)	–0.004(1)	–0.0074(9)
C(3)	2*i*	0.4540(5)	0.1881(3)	–0.0101(2)	0.102(3)	0.025(1)	0.021(1)	–0.011(1)	–0.009(1)	–0.0098(9)
C(4)	2*i*	0.2655(6)	0.2269(4)	0.0337(3)	0.100(3)	0.045(2)	0.030(1)	–0.040(2)	–0.017(1)	–0.010(1)
C(5)	2*i*	0.1958(4)	0.3149(3)	0.1193(2)	0.047(1)	0.045(2)	0.030(1)	–0.024(1)	–0.007(1)	–0.014(1)
C(6)	2*i*	0.3186(3)	0.3611(2)	0.1634(2)	0.032(1)	0.022(1)	0.0155(8)	–0.0076(8)	–0.0043(7)	–0.0064(7)
C(7)	2*i*	0.2323(3)	0.4695(2)	0.2425(2)	0.0251(9)	0.025(1)	0.0177(8)	–0.0052(8)	–0.0028(7)	–0.0083(7)
C(8)	2*i*	0.2768(3)	0.4355(2)	0.3795(2)	0.0221(8)	0.0213(9)	0.0156(8)	–0.0073(7)	–0.0017(6)	–0.0070(7)
C(9)	2*i*	0.3830(3)	0.2944(2)	0.4402(2)	0.0243(9)	0.023(1)	0.0193(8)	–0.0080(7)	0.0001(7)	–0.0092(7)
C(10)	2*i*	0.4258(3)	0.2583(2)	0.5675(2)	0.0250(9)	0.0195(9)	0.0197(9)	–0.0058(7)	–0.0045(7)	–0.0036(7)
C(11)	2*i*	0.3641(3)	0.3611(3)	0.6380(2)	0.035(1)	0.030(1)	0.0172(9)	–0.0082(9)	–0.0057(8)	–0.0079(8)
C(12)	2*i*	0.2581(3)	0.5009(2)	0.5804(2)	0.033(1)	0.026(1)	0.0198(9)	–0.0059(9)	–0.0039(8)	–0.0109(8)
C(13)	2*i*	0.2117(3)	0.5410(2)	0.4514(2)	0.0223(8)	0.021(1)	0.0172(8)	–0.0070(7)	–0.0009(7)	–0.0067(7)
C(14)	2*i*	0.0256(3)	0.7965(2)	0.4432(2)	0.0245(9)	0.022(1)	0.0269(9)	–0.0068(8)	–0.0009(7)	–0.0109(8)
C(15)	2*i*	–0.0693(3)	0.9419(2)	0.3504(2)	0.029(1)	0.023(1)	0.032(1)	–0.0019(8)	–0.0042(8)	–0.0119(9)

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